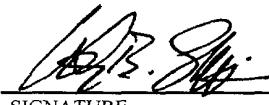


FORM PTO-1390 (REV 10-95)	U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. §371			MERCK 2257
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE		U.S. APPLICATION NO (If known, see 37 CFR §1.5)
PCT/EP99/09485	3 DECEMBER 1999		09 / 857602
TITLE OF INVENTION		PRIORITY DATE CLAIMED	
METHOD FOR PRODUCING MOLTEN SALTS WITH AN EXTRUDER AND USE OF THE MOLTEN SALTS		15 DECEMBER 1998	
APPLICANT(S) FOR DO/EO/US			
OHREM, Hans-Leonhard, et al.			
<p>Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:</p> <ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. §371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. §371. 3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(1). 4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. §371(c)(2)) <ul style="list-style-type: none"> a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau). b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. §371(c)(2)). 7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3)) <ul style="list-style-type: none"> a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau). b. <input checked="" type="checkbox"/> have been transmitted by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)). 9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)). 10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)). <p>Items 11. to 16. below concern document(s) or information included:</p> <ol style="list-style-type: none"> 11. <input type="checkbox"/> An Information Disclosure Statement under 37 C.F.R. §§1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> Other items or information: 			

U.S. APPLICATION NO. (if known) (37 CFR §1.15) 097857602		INTERNATIONAL APPLICATION NO. PCT/EP99/09485	ATTORNEY'S DOCKET NUMBER MERCK 2257
17. <input checked="" type="checkbox"/> The following fees are submitted:		CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR §1.492 (a) (1) - (5)):			
Search Report has been prepared by the EPO or JPO.....		\$860.00	
International preliminary examination fee paid to USPTO (37 CFR §1.482).....		\$690.00	
No international preliminary examination fee paid to USPTO (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2)).....		\$710.00	
Neither international preliminary examination fee (37 CFR §1.482) nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO.....		\$1000.00	
International preliminary examination fee paid to USPTO (37 CFR §1.482) and all claims satisfied provisions of PCT Article 33(2)-(4).....		\$100.00	
ENTER APPROPRIATE BASIC FEE AMOUNT =		\$860.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 C.F.R. §1.492(e)).		<input type="checkbox"/> 20	<input type="checkbox"/> 30
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	10 - 20 =	0	x \$ 18.00 \$0.00
Independent claims	1 - 3 =	0	x \$ 80.00 \$0.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)		+ \$ 270.00	
TOTAL OF ABOVE CALCULATIONS =		\$860.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be filed (Note 37 C.F.R. §§1.9, 1.27, 1.28).			
SUBTOTAL =		\$860.00	
Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 C.F.R. §1.492(f)).		<input type="checkbox"/> 20	<input type="checkbox"/> 30
TOTAL NATIONAL FEE =		\$860.00	
Fee for recording the enclosed assignment (37 C.F.R. §1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. §§3.28, 3.31). \$40.00 per property.			
TOTAL FEES ENCLOSED =		\$860.00	
		Amount to be refunded:	
		charged:	
<p>a. <input checked="" type="checkbox"/> A check in the amount of <u>\$860.00</u> to cover the above fees is enclosed.</p> <p>b. <input type="checkbox"/> Please charge my Deposit Account No. <u>13-3402</u> in the amount of <u>\$</u> to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>13-3402</u>. A duplicate copy of this sheet is enclosed.</p>			
<p>NOTE: Where an appropriate time limit under 37 C.F.R. §§1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p>			
SEND ALL CORRESPONDENCE TO: Customer Number 23,599			
PATENT TRADEMARK OFFICE			
 <u>23599</u>			
 SIGNATURE			
<u>Harry B. Shubin</u> NAME			
<u>32,004</u> REGISTRATION NUMBER			
Filed: 8 JUNE 2001			
AJZ(HBS):jmm			

IN THE UNITED STATES DESIGNATED/ELECTED OFFICE

International Application No. : PCT/EP99/09485
International Filing Date : 3 DECEMBER 1999
Priority Date(s) Claimed : 15 DECEMBER 1998
Applicant(s) (DO/EO/US) : OHREM, Hans-Leonhard, et al.

Title: METHOD FOR PRODUCING MOLTEN SALTS WITH AN EXTRUDER AND
USE OF THE MOLTEN SALTS

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

SIR:

Although the claims of the above application were amended under Article 34 during the International Phase, applicants respectfully request that the claims as originally filed by examined and this preliminary amendment is based thereon.

Prior to calculating the national fee, and prior to examination in the National Phase of the above-identified International application, please amend as follows:

IN THE CLAIMS:

3. (Amended) Process according to Claim 1, characterized in that the reaction is carried out under reduced pressure, atmospheric pressure or superatmospheric pressure in the presence of atmospheric oxygen or, if desired, under a protective-gas atmosphere.

REMARKS

The purpose of this Preliminary Amendment is to eliminate multiple dependent claims in order to avoid the additional fee. Applicants reserve the right to reintroduce claims to canceled combined subject matter.

Respectfully submitted,



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AJZ(HBS):jmm

FILED: 8 JUNE 2001

VERSION WITH MARKINGS TO SHOW CHANGES MADE

Claim 3 has been amended as follows:

3. (Amended) Process according to Claims 1 and 2, characterized in that the reaction is carried out under reduced pressure, atmospheric pressure or superatmospheric pressure in the presence of atmospheric oxygen or, if desired, under a protective-gas atmosphere.

110RTS

09/857602

JC18 Rec'd PCT/PTO 08 JUN 2001

Process for the preparation of salt melts using an extruder, and the use thereof

5 The invention relates to a process for the preparation of salt melts and mixtures thereof by means of an extruder in which the starting materials are melted and brought to reaction, and the reaction products are subsequently passed through a tower or column containing alkali metal salt.

10

Extruders are traditionally employed for melting and mixing polymeric materials, such as plastics. The extruders can also be used as reactors for polymerizations. Anionic polymerization in an extruder is described, for example, for caprolactam to nylon 6 (B. VanBuskirk, M.K. Akkapeddi, Polym. Prepr. Vol. 29, 557 (1988)). The advantage of using an extruder rather than a stirred-tank reactor for polymerization reactions is that it is easier to process high-viscosity substances. The requirement for thorough mixing of all substances participating in the reaction and good heat transfer can also be achieved optimally by using an extruder (G. Menges et al., New Polym. Mater., Proc. Int. Semin., 129-148 (1987)). In EP 813 904, pharmaceutical active ingredients are prepared by means of extruders. Here, pharmaceutical active ingredients carrying acid groups are reacted with a base in the melt.

30 Melts of salts, such as, for example, NaAlCl_4 , have various areas of application. Salt melts can be employed as storage medium in heat stores, as heat-transfer agents, for example in heating baths, for covering and purifying molten metals, for electro-
35 coating of high-melting materials or as melt electrolyte in primary batteries, as described in GB 2,046,506. A further possible application of these salts is in re-chargeable sodium batteries. The salts are employed in batteries which have operating
40 temperatures of between 130°C and 200°C (K.M. Abraham,

D.M. Pasquariello, J. Electrochem. Soc., Vol. 137.
1189-1190 (1990)).

5 DE 3419279 describes an electrochemical cell in which
the cathode matrix is impregnated with a
sodium/aluminium halide salt melt electrolyte.

10 A relatively new area of application is the "ZEBRA
battery". This high-temperature cell consists of an
electrode of liquid sodium, a beta-aluminium
electrolyte and an electrode of transition-metal
chloride in an NaAlCl_4 melt (B. Cleaver,
V.S. Sharivker, J. Electrochem. Soc., Vol. 142, 3409-
3413 (1995)).

15

20 DE 3718920 describes the preparation of salt melts by
adding a pure metal and an alkali metal halide to the
melt. The reaction cell is operated above the melting
point of the salt melt. In the working example, the
alkali metal halide is NaCl , the molten alkali metal is
sodium, and the separator is beta-aluminium oxide.
Owing to the use of pure sodium, special safety
precautions, such as working under a protective-gas
atmosphere, must be taken. The reactions must take
25 place in separate cells, since poisoning of the
separator by the by-product AlHal_3 formed must be
prevented.

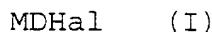
30 For the preparation of the alkali metal halogen
aluminates, the reaction of corresponding aluminium
halides and alkali metal halides in a sealed tube is
described (Friedmann, Taube, J. Am. Chem. Soc., 72,
2236-2243 (1950)). In this process, an increase in
pressure to 6 - 7 atmospheres is observed, which
35 results in problems (FR 2168912). The apparatuses must
be fitted with the appropriate safety precautions.

All the processes disclosed hitherto for the
preparation of salt melts operate batchwise. A batch

procedure has some severe disadvantages compared with a continuous preparation process. During a batch change, the apparatus must be opened. The product can then be contaminated by the oxygen from the ambient air, water and dust. The batch change results in downtime of the plants and thus in a reduced space-time yield. An effective discontinuous process requires large apparatuses. The start-up process requires correspondingly more energy and time. It has been found that, in particular during start-up of the plants, impurities can be introduced into the process. FR 2168912 describes a complex purification process for alkali metal halogen alumimates. The 2-step purification process consists of oxygen treatment for degrading the organic impurities and aluminium treatment for precipitating iron and heavy metals. The aluminium treatment must be carried out under a nitrogen or argon atmosphere.

20 The object of the invention is to provide a continuous process for the preparation of pure salt melts which excludes the disadvantageous effects of the environment, minimizes the energy requirement and facilitates an optimum space-time yield. A further 25 object is to make large amounts of salt melts available in the shortest possible time.

30 The object according to the invention is achieved by a process for the preparation of salt melts, and mixtures thereof, of the general formula



in which

35 M is Li, Na, K, Rb or Cs,

D is Al, Ga, In or Tl, and

Hal is F, Cl, Br or I,

characterized in that the starting materials, a metal halide and an alkali metal halide, are melted and
5 brought to reaction continuously or, if desired, discontinuously in a heatable extruder with forced conveying, and the reaction products are subsequently passed through a tower or column containing alkali metal salt.

10

The process products are suitable for use as melt electrolyte in electrochemical cells, as storage medium in heat stores, as heat-transfer agent, for example in heating baths, for covering and purifying molten
15 metals, for electrocoating of high-melting materials or as melt electrolyte in rechargeable sodium batteries and primary batteries.

Surprisingly, it has been found that the starting
20 materials can be processed by means of an extruder. It is possible to prevent the feared contamination of the product by abrasion by means of a suitable choice of material and residence time.

25 It has been found that forced conveying in the extruder by pumps can be omitted in this process, which considerably reduces the susceptibility of the process to faults.

30 Any extruder which appears suitable to the person skilled in the art can be used for the process. Particularly suitable extruders are single-screw extruders, multiscrew extruders with co-rotating and counterrotating screws, vented extruders, planetary-gear extruders, ram extruders and disc extruders. For
35 the processing of salts in extruders, the hardness of the substances employed and their chemical properties represent particular problems which have hitherto stood in the way of implementation. In general, the extruder

000000000000000000000000

is made of steel. This material would be badly damaged by corrosion and abrasion during processing of salts.

It has been found that corrosion can be greatly reduced
5 in extruders whose essential components are made of
nickel alloys. It has furthermore been found that the
metal parts of the extruder which come into contact
with the salts or their melts can be protected against
corrosive and abrasive damage by surface coatings with
10 materials known to the person skilled in the art, such
as PTFE/PFA, enamel or ceramic materials. In order to
reduce abrasion, an additional bearing can be installed
at the head of the screw.

15 Surprisingly, it has been found that the installation
of screw elements with a reversed flow direction allows
a completely molten and homogeneous product to be
obtained in spite of a very short average residence
time of the material in the extruder of a few seconds.

20 The reaction in the extruder can be carried out in the
presence of atmospheric oxygen or, if desired, under a
protective-gas atmosphere (for example nitrogen, CO₂ or
noble gases) at a reduced pressure, atmospheric pres-
25 sure or even at superatmospheric pressure at
temperatures of from 50°C to 800°C (at atmospheric
pressure). When working under superatmospheric pressure
or reduced pressure, the melting points of the salts
shift correspondingly and the heating stages of the
30 extruder are modified correspondingly.

Processing should be carried out below the sublimation
temperature of the starting materials. The reaction is
preferably carried out at elevated temperatures, since
35 the solubility of the salts is significantly better
under such conditions.

During processing of the salts in the extruder, a
specific choice of temperature in the heating stages

allows an optimum temperature programme to be set during the process.

In order to carry out the process, the aluminium halide
5 employed is a fluoride, chloride, bromide or iodide, or mixtures thereof. Suitable alkali metal salts are lithium, sodium, potassium, rubidium or caesium fluoride, chloride, bromide or iodide, or mixtures thereof.

10 A general example of the invention is explained in greater detail below and is shown in the drawing. Fig. 1 shows a heatable extruder 1 with a solids metering device 2 and downstream column or tower 3.

15 In order to prepare salts conforming to the formula
(I), and mixtures thereof, the starting materials can
be fed to the extruder separately via solids metering
devices 2. The starting materials can also be fed in,
20 premixed in the same ratio, via a single metering
device. The extruder can also be filled under an inert
gas. An extruder with forced conveying pushes the salt
bed forwards in the screw channel with screw speeds of
between 1 rpm and 75 rpm. The screw geometry can have
25 an l/d ratio of between 3 and 25. In the heating zones
of the jacket 1, the melting temperatures for various
starting materials and end products can be set. In the
final quarter, it is possible to use screw elements
which cause back-mixing. This increases the residence
30 time in this zone, and as yet unmelted salts are mixed
with liquid melt.

The low-viscosity melt produced by the process is fed to a column or tower 3 containing the corresponding alkali metal salt. The melt is passed through the alkali metal salt in order to react residual amounts of unreacted metal halide.

The conveying pressure built up by the extruder can be utilized for the transport of the melt to and through the tower or column.

5 The example given below is given in order to better illustrate the present invention, but is not suitable for restricting the invention to the features disclosed therein.

10

Examples

Example 1:

Preparation of NaAlCl₄

15

In order to prepare 1 kg/h of NaAlCl₄, 373.8 g/h of NaCl is fed to an extruder via a solids metering device and 626.2 g/h of AlCl₃ is fed to the extruder via a further solids metering device. A twin-screw extruder screw with forced conveying pushes this salt bed forwards in the screw channel at a screw speed of 25 rpm. The various heating zones of the jacket can be adapted so that the salt is brought to the melting temperature of the salt during the stretch from the feed opening to the discharge zone. In the preparation of NaAlCl₄, a temperature of 182°C is set in the feed. The final quarter contains screw elements which have a reversed conveying direction, thus increasing the residence time of the mixture in this zone. Unmelted salts are mixed with liquid melt in order to facilitate better heat transfer.

The low-viscosity melt formed is fed via a pipeline to a tower containing a bed of sodium chloride granules. 35 In the tower, residues of unreacted AlCl₃ are converted to NaAlCl₄. The conveying pressure of the extruder is sufficient to transport the melt to and through the tower without an additional pump device.

PATENT CLAIMS

1. Process for the preparation of salt melts, and mixtures thereof, of the general formula

5

MDHal (I)

in which

10 M is Li, Na, K, Rb or Cs,

D is Al, Ga, In or Tl, and

Hal is F, Cl, Br or I,

15

characterized in that the starting materials, a metal halide and an alkali metal salt, are fed continuously or, if desired, discontinuously via a solids metering device and melted and brought to reaction in a heatable 20 extruder (1) with forced conveying, and the reaction products are subsequently passed through a tower or column (3) containing alkali metal salt.

25 2. Process according to Claim 1, characterized in that the starting materials are melted and brought to reaction in a heatable extruder (1) with forced conveying at a temperature between 50°C and 800°C.

30 3. Process according to Claims 1 and 2, characterized in that the reaction is carried out under reduced pressure, atmospheric pressure or superatmospheric pressure in the presence of atmospheric oxygen or, if desired, under a protective-gas atmosphere.

35 4. Process according to Claim 1, characterized in that the salt bed is processed in an extruder whose parts which come into contact with the salts or melts thereof are made of an Ni alloy or of metals coated with PTFE/PFA, enamel or ceramic materials and which

has a screw channel having an l/d ratio of between 3 and 25, at screw speeds of between 1 rpm and 75 rpm.

5. Use of salts of the general formula (I) according
5 to Claim 1 as melt electrolyte in electrochemical cells
and batteries.

6. Use of salts of the general formula (I) according
10 to Claim 1 as melt electrolyte for rechargeable sodium
batteries and primary batteries.

7. Use of salts of the general formula (I) according
to Claim 1 as storage medium in heat stores.

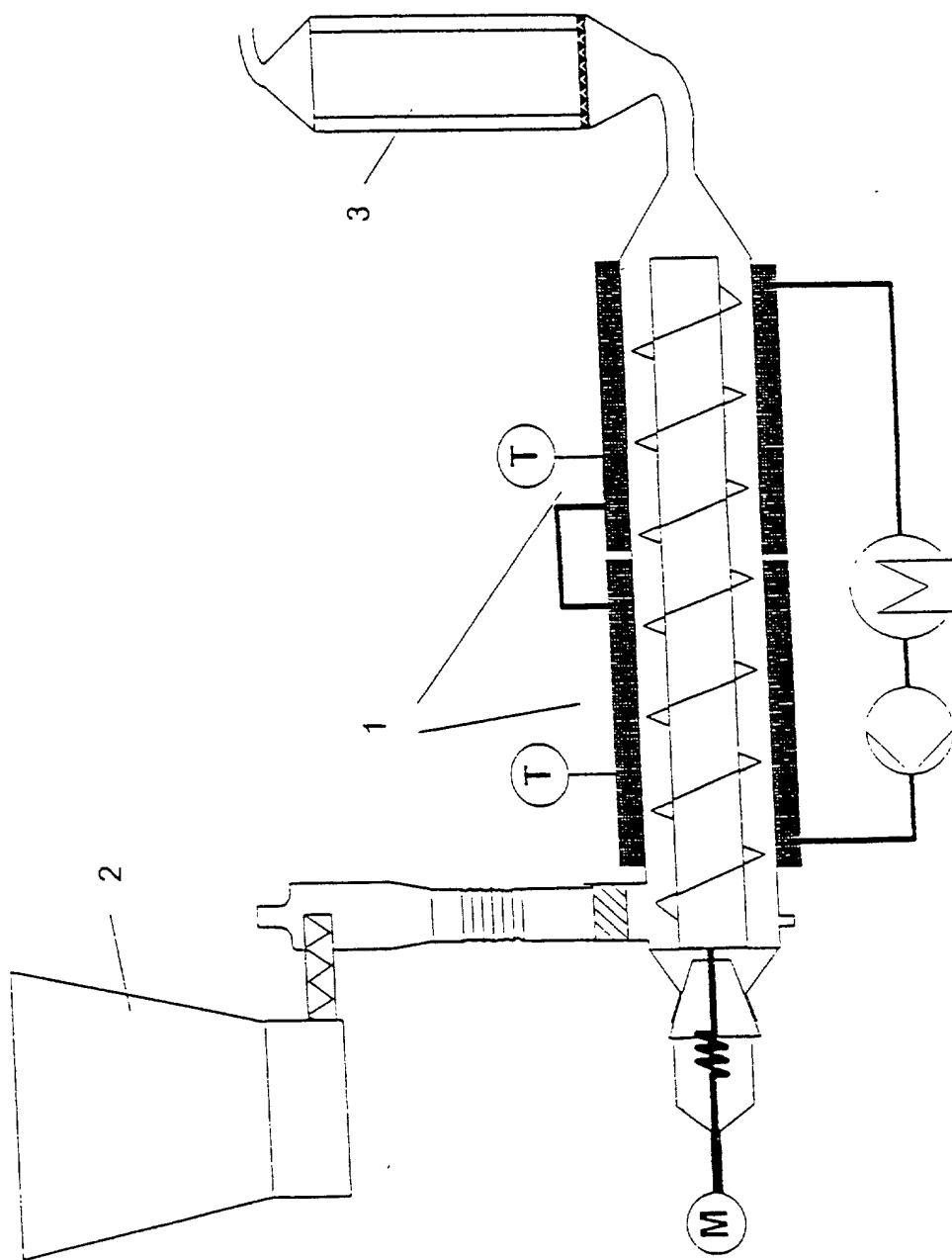
15 8. Use of salts of the general formula (I) according
to Claim 1 as heat-transfer agent.

9. Use of salts of the general formula (I) according
to Claim 1 for covering and purifying molten metals.

20 10. Use of salts of the general formula (I) according
to Claim 1 for electrocoating of high-melting
materials.

1/1

FIG. 1



Docket No.
Merck

**Declaration and Power of Attorney For Patent Application
English Language Declaration**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Method for producing molten salts with an extruder and use of the molten salts

the specification of which

(check one)

is attached hereto.
 was filed on 03.12.1999 as United States Application No. or PCT International Application Number PCT/EP99/09485
and was amended on _____
(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

<u>198 57 788.5</u> (Number)	<u>Germany</u> (Country)	<u>15.12.1998</u> (Day/Month/Year Filed)
<hr/> (Number)	<hr/> (Country)	<hr/> (Day/Month/Year Filed)
<hr/> (Number)	<hr/> (Country)	<hr/> (Day/Month/Year Filed)

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

I hereby claim the benefit under 35 U.S.C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112. I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C.F.R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)
(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint

16
 I. William Millen (Reg. No. 19,544)
 John L. White (Reg. No. 17,746)
 Anthony J. Zelano (Reg. No. 27,969)
 Alan E.J. Branigan (Reg. No. 20,565)
 John R. Moses (Reg. No. 24,983)
 Harry B. Shubin (Reg. No. 32,004)
 Brion P. Heaney (Reg. No. 32,542)
 Richard J. Traverso (Reg. No. 30,595)

John A. Sopp (Reg. No. 33,103)
 Richard M. Lebovitz (Reg. No. 37,067)
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 Catherine M. Joyce (Reg. No. 40,668)
 James T. Moore (Reg. No. 35,619)
 James E. Ruland (Reg. No. 37,432)
 Nancy Axelrod (Reg. No. 44,014)
 Jennifer J. Branigan (Reg. No. 40,921)

to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

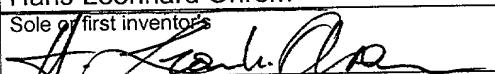
Send Correspondence to: MILLEN; WHITE; ZELANO & BRANIGAN, P.C.
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 Arlington, VA 22201

Direct Telephone Calls to: (name and telephone number)

1-00
 Full name of sole or first inventor

Hans-Leonhard Ohrem

Sole or first inventor's signature



Date

April 11, 2001

Residence

Gutenbergstrasse 6a, 64342 Jugenheim Germany



Citizenship

German

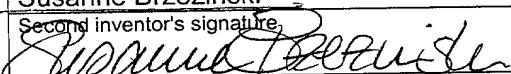
Post Office Address

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2-00
 Full name of second inventor, if any

Susanne Brzezinski

Second inventor's signature



Date

April 11, 2001

Residence

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Citizenship

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Post Office Address

c/o Merck KGaA, Frankfurter Str. 250, 64293 Darmstadt, Germany